

TO BE ADDRESSED

Dear Authors,

You have addressed all the concerns of the Reviewers, except the following point:

“p3 Section 3.2 and Figure 2: How thick are these GO layers, since since the (100) Si substrate is not visible on the XRD patterns. I would expect to see a strong peak for the Si”.

This point also relates to point 2 of the 2nd Reviewer, i.e. “at a first glance, it is not immediately clear that the SEM images show a highly dispersed ‘coating’ of GO particles. It can be interpreted as a very smooth, continuous GO film with particles on top. The discussion section comments on the highly non-uniformed nature of the coating, but it is suggested that this is also mentioned in section 3.1”.

Clearly there is no SI peak visible in the XRD patterns, and since it appears from Figure 1 that the “films” consist of islands of GO particles, this comment/question of the Reviewer needs to be addressed before the article can be accepted for publication.

I discussed the matter with prof Mike Lee, and his comments were the following:

- a. He questions that you were able to measure a 15 nm thick layer with cross-sectional SEM. (If he is wrong, please send us a micrograph).
- b. For such a thin film, the XRD should have clearly shown the Si peak (between 20 – 30 2q degrees). Remember, the X-rays have a deep penetration depth.
- c. Section 2: the SEM microscope is a JEOL, not a JOEL.
- d. A quick calculation shows that the magnification of the micrographs in Figure 2 to be ~ 7,000x , not “100 times” as stated on page 3 under Section 3.1.

(He also added that it would have been better to have used e.g. 2 kV accelerating voltage, rather than the 10 kV used, but that is just a comment).

None the less, the main concern remains the Si peak not seen in the XRD images. Please address this issue (the layers are not perhaps 15 micron thick, rather than 15 nm?)

Yours sincerely,
Japie

Editor: SAIP 2018 Conference Proceedings

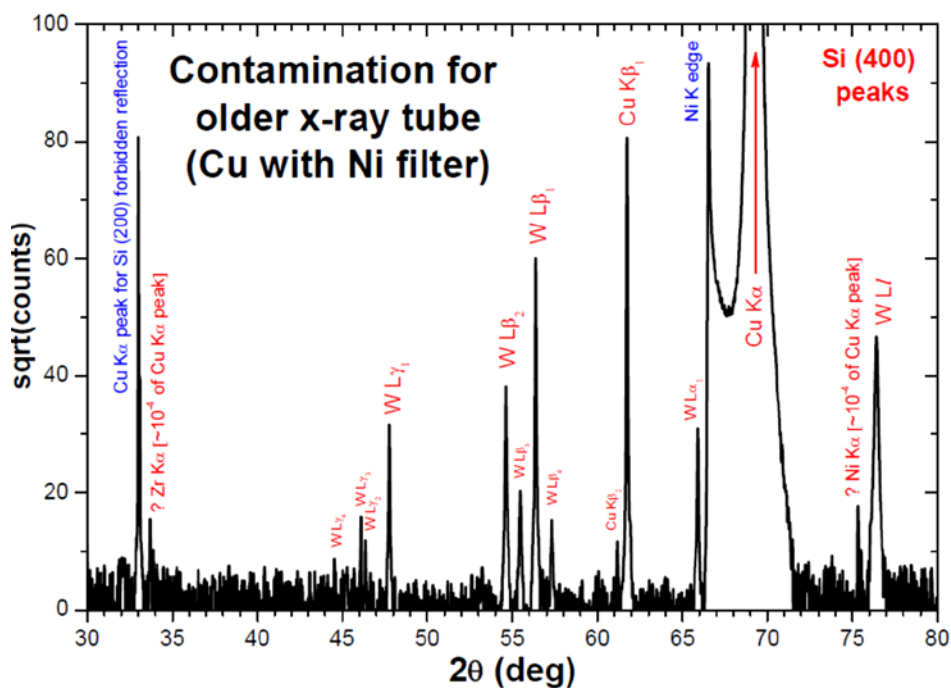
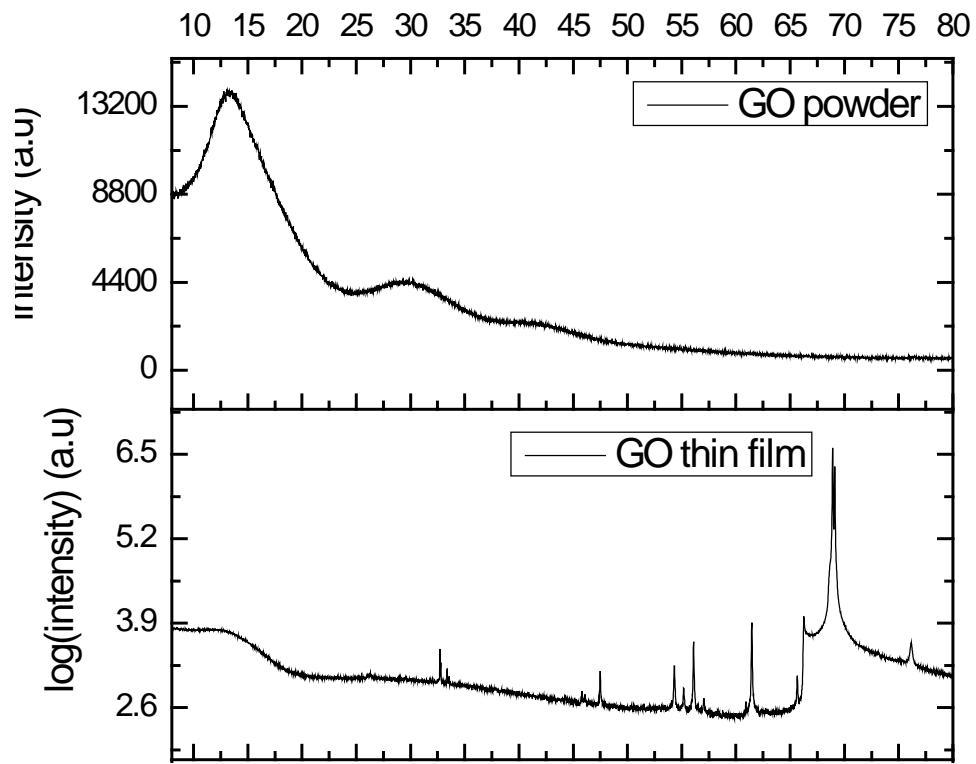
RESPONSE

We thank the editor for these comments, which have allowed us to improve the paper.

1. For XRD, regarding the absence of the peak corresponding to the (100) plane of the Si substrate:

(a) When samples were rotated during the XRD measurements, the 400 peak of the Si substrate was indeed observed near $2\theta = 68^\circ$, and was very intense. An example is shown below, where the bottom panel is plotted on a log scale so that both very large and also small peaks can be viewed on the same graph. In addition to the huge Si 400 peak, the forbidden Si 200 peak near 33° , several orders of magnitude smaller, is also visible. There are also numerous other small sharp lines, e.g. near 62° due to the weak Cu $K\beta$ X-rays (which are reduced, but not eliminated, by the Ni filter) and a series of many lines between 45° and 58° which arise from weak characteristic tungsten X-rays of different wavelengths (various W $L\beta$ and $L\gamma$) all diffracting from Si (400) planes. This arises when tungsten from the cathode

contaminates the copper anode where X-rays are generated over time and we have replaced our X-ray source in the past when these peaks become problematic.



(b) When samples are NOT rotated, these peaks are sometimes seen, but often they are no longer observed. This is due to a slight mis-orientation of the substrate in the holder. This effect can also occur

when the substrate is intentionally cut slightly off-axis, although that was not the case in our study. This scenario is addressed nicely in a discussion on the website “ResearchGate” reproduced below where, in particular, in the last paragraph it is explained: “*You can use this effect to your benefit by misaligning your substrate crystal plane on purpose, using an omega offset. Thereby making the normally huge substrate peak invisible and increasing the visibility of your thin film crystal structures.*” We believe this is the case in our study, with the offset caused by how the sample was placed in the diffractometer. In this scenario, the substrate peaks are avoided even if the film is very thin for even not continuous.

ResearchGate

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Question Asked June 2, 2015

Murali Krishnan
14.95 · National Aerospace Laboratories

Why substrate peaks are missing in some XRD spectrum?
I have taken the XRD spectra of series of samples with same structure deposited on MgO(100) substrate and annealed at four different temperatures. Surprisingly, two of the samples annealed at higher temperatures are having the diffraction peak corresponding to substrate. Whereas, for the other two annealed at lower temperature the MgO peak is not seen!! Could anybody please explain the reason for th

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Harry J. Wondergem
added an answer

June 8, 2015

The strange behavior of your MgO-2.0.0 peak can be caused by the miscut angle of your MgO crystals. In single crystalline substrates, cut from a larger crystal, the sample surface is usually not exactly parallel to the aimed for crystal plane. This miscut angle can easily be a few degrees. When you perfectly align the sample surface on your diffractometer, the crystal will therefore be miss aligned. When the normal to the crystal plane is in the diffraction plane, the peak will be invisible if the miscut angle is larger than the divergence angle. If the normal to the crystal plane is outside the diffraction plane your substrate peak will be invisible when the miscut angle is larger than the axial divergence. As the axial divergence is usually much larger than the in-plane divergence it is possible to either see or not see the substrate peak depending on how your sample is put in the diffractometer.

You can find the miscut angle of your substrate by making a pole figure of the MgO-2.0.0 peak, or make a series of rocking curves of that peak while rotating your sample in between measurements.

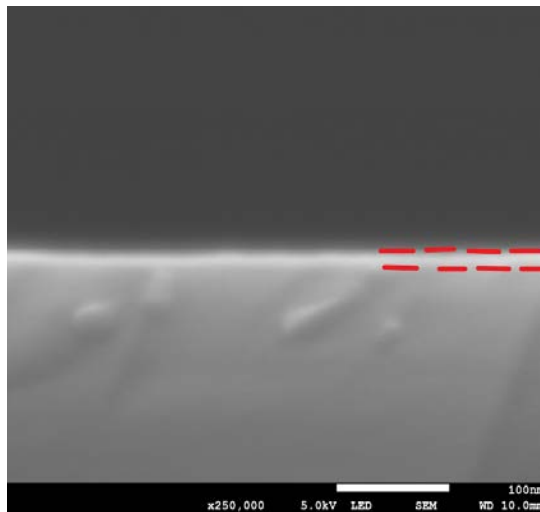
You can use this effect to your benefit by misaligning your substrate crystal plane on purpose, using an omega offset. Thereby making the normally huge substrate peak invisible and increasing the visibility of your thin film crystal structures.

(c) To address this, we added the following sentence to section 3.2 of the paper: “Peaks from the silicon substrate were avoided by not rotating the sample, which was placed at a slight mis-orientation perpendicular to the diffractometer rotation axis.”

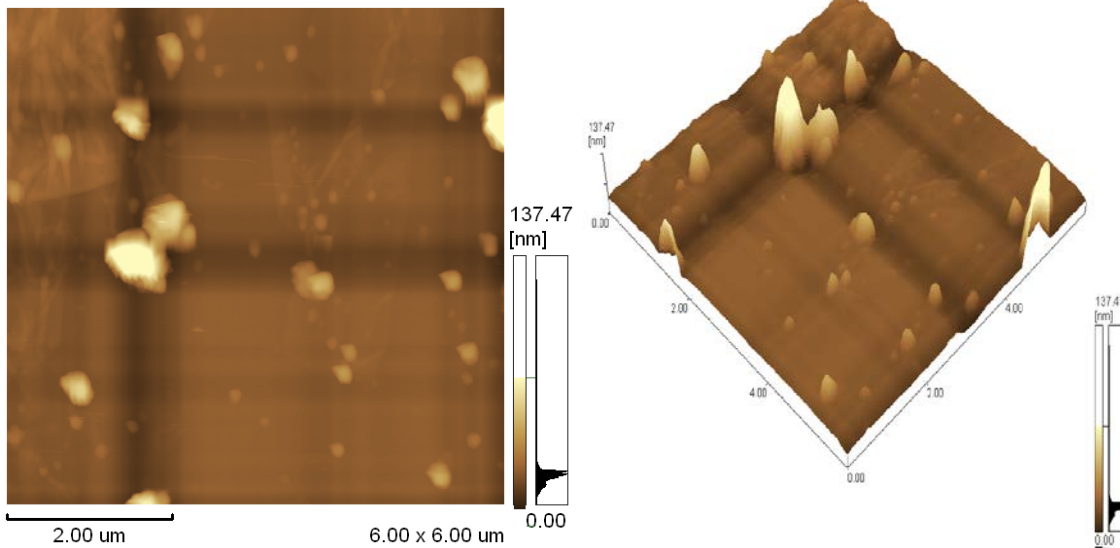
2. Regarding the thickness:

(a) We show below the SEM image which was originally used to obtain the estimate of 15 nm. However, we have reconsidered and agree with Prof Lee that this image and the value given DO NOT represent the film thickness. Considering the higher magnification plan view SEM images in the paper which now

clearly show that the “films” consist of islands of GO particles, we have now performed atomic force microscopy (AFM) to obtain height information of the particles on the surface.



(b) The new AFM images are shown below. From its vertical scale we estimate the size of the GO structures to be of the order of 100 nm and have added the AFM image and this estimate to the paper in section 3.1 as follows: “The surface topography of this film was examined using AFM as shown in Figure 2, where the height of GO clusters was estimated to be about 100 nm.” We retain the word “film” as a description of the pin coated material, but have deleted the phrase “thin film” throughout to article due to the incomplete coverage of the layer.



3. We corrected JOEL to JEOL. Regarding the magnification (e.g. 7000x) of the SEM images, we acknowledge this was an error which occurred when we replaced the SEM images during the first revision. Since the magnification depends on the size viewed or printed, we have deleted this information and rely only on the scale bar in the images to represent the magnification.
